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(54) Process for bleaching of lignocellulose-containing pulp

Verfahren zum Bleichen von Lignocellulose enthaltendem Zellstoff

Procédé pour le blanchiment de pâte à papier contenant de la lignocellulose

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Description

The present invention relates to a process for delignification and bleaching of chemically digested lignocellulose-containing pulp. The pulp is bleached at a pH of between about 1 and about 6, whereupon a water-soluble compound containing an alkaline earth metal is added at a pH of between about 1 and about 7 before the pulp is treated with a chlorine-free bleaching agent. The initial acidic treatment removes the trace metals of the pulp, whereas the subsequent addition of alkaline earth metal ions in aqueous solution returns the ions to the positions in the pulp where they have a particularly beneficial effect on the preservation of the cellulose chains and, consequently, on the viscosity, as well as on the consumption of bleaching agent in the subsequent bleaching step. After the treatment according to the invention, the pulp may be finally bleached to the desired brightness, suitably with a chlorine-free bleaching agent, such as ozone, to completely avoid formation and discharge of AOX.

Background

In the production of chemical pulp of high brightness, wood chips are first cooked to separate the cellulose fibres. Part of the lignin holding the fibres together is thus degraded and modified, such that it can be removed by subsequent washing. However, in order to obtain sufficient brightness, more lignin has to be removed, together with brightness-impairing (chromophoric) groups. This is frequently effected by delignification with oxygen, followed by bleaching in several stages.

For environmental reasons, it has become increasingly common to treat chemical pulp with chlorine-free bleaching agents already in the first bleaching steps. The big advantage is the drastic reduction in the discharges of chlorinated organic substances detrimental to the environment, owing to the combined effect of a smaller amount of chlorine-containing bleaching agents and lower content of lignin, which is the organic substance primarily reacting with the chlorine.

It is known to use chlorine-free bleaching agents, such as hydrogen peroxide, peracetic acid or ozone, already in the prebleaching. However, the delignification and consumption of the bleaching agent become less effective than with chlorine-containing bleaching agents, unless the pulp is pretreated. Thus, a hydrogen peroxide treatment in an alkaline environment is disturbed by the presence in the pulp of ions of certain metals, such as Mn, Cu and Fe. These metal ions cause degradation of hydrogen peroxide, thereby reducing the efficiency of the peroxide treatment and increasing the consumption of peroxide. According to CA-A-1,206,704, this can be counteracted by pretreating the pulp with an acid, such as sulphuric acid or nitric acid, whereby the concentration of all types of metal ions is reduced. However, by this treatment also metal ions, for example Mg, which are advantageous to the peroxide treatment disappear, which ions stabilize the peroxide and increase the selectivity of the peroxide.

CA-A-575,636 discloses the addition of magnesium sulphate to stabilize alkaline peroxide solutions. However, the addition is made directly to the bleaching liquor and in alkaline environment insoluble magnesium hydroxide precipitates. Furthermore, US-A-4,222,819 discloses the addition of magnesium ions to acidic peroxide solutions, but also in this case the addition is made directly to the bleaching liquor. None of the related methods makes possible diffusion of the magnesium ions into the pulp to such an extent, that a pulp of high brightness and strength can be obtained. US-A-3,507,744 relates to a bleaching process for wood pulp, where the pulp is contacted with an alkaline solution containing hydrogen peroxide and further a pyrophosphate to stabilize the peroxide. The aim of the process is to attain high brightness in a single bleaching step without a substantial loss in yield. The process can be preceded by a pre-treatment in one or more steps, for example acid treatment and/or addition of barium or calcium salts.

DE-A-2022866 describes the use of acid waste liquids deriving from pulp bleaching, with Cl₂/ClO₂, to carry out an acid pretreatment, i.e. a washing stage. Since the waste liquids derives from a bleaching stage where Cl₂/ClO₂ are used as active bleaching chemicals, there will be no, or a unsignificant amount of Cl₂/ClO₂ remaining in the waste liquids.

J.P. Casey "Pulp and Paper Chemistry and Chemical Technology" 3rd edition, vol. 1, 1980, John Wiley & Sons, New York, p 651 ("Additional Chemicals used with Peroxide"), discloses that sodium silicate, magnesium sulphate or calcium may be added in the hydrogen peroxide step in order to inactivate metal ions and to improve the bleaching with hydrogen peroxide.

The invention

The invention provides a process in which lignocellulose-containing pulp is treated under the conditions disclosed in the claims, whereby the metal ions harmful to the subsequent bleaching are effectively removed and the profile of alkaline earth metals is restored before the pulp is bleached in a chlorine-free bleaching step.

The invention relates to a process for bleaching of chemically digested lignocellulose-containing pulp as described in claim 1, wherein the pulp is bleached at a pH in the range from about 1 up to about 6, whereupon a Mg compound

is added at a pH in the range from about 1 up to about 7 and in an amount of from about 0.01 kg, suitably 0.5 kg, up to about 10 kg/ton of dry pulp, calculated as magnesium, and that the pulp subsequently is treated with a chlorine-free bleaching agent, i.e. hydrogen peroxide.

Acid treatment is an effective process to eliminate metal ions from lignocellulose-containing pulps. At the same time it is known, that ions of alkaline earth metals, especially when in their original positions in the pulp, have a positive influence on the selectivity of the delignification as well as on the stability and consumption of chlorine-free bleaching agents, such as peroxides, ozone and oxygen. The present process presents an economic solution to the problem of creating a suitable trace-metal profile for the subsequent chlorine-free bleaching, in that non-desirable metal ions are eliminated while supplied ions of magnesium essentially recover the positions in the vicinity of the cellulose chains previously occupied by ions of alkaline earth metals. This is achieved by adding the magnesium compound at such a pH and such a temperature that the compound is dissolved in water, thus enabling the diffusion required to obtain the intended effect. Furthermore, an advantage of the present process is that the pH adjustment between the treatment with acid and addition of magnesium ions becomes very limited or may be left out altogether, which is advantageous to process technique and economy.

Chlorine-free bleaching agents include inorganic peroxide compounds, such as hydrogen peroxide and sodium peroxide, organic peroxide compounds, such as peracetic acid, as well as ozone, oxygen and sodium dithionite. Suitably, hydrogen peroxide (P), oxygen (O) and ozone (Z) are used in a sequence or mixture. Preferably, use is made of mixtures of hydrogen peroxide and oxygen (PO). The sequence P-Z or (PO)-Z are especially preferred.

In the treatment with hydrogen peroxide in an alkaline environment, pH is suitably adjusted by adding to the pulp an alkali or an alkali-containing liquid, such as sodium carbonate, sodium hydrogen carbonate, sodium hydroxide, oxidized white liquor or magnesium hydroxide slurry. Suitably, the magnesium hydroxide slurry is taken from the chemical handling system in the production of sulphite pulp with magnesium as base, i.e. magnetite pulp.

The acid bleaching step suitably is carried out with an acid and the respective bleaching agent. The acids used are inorganic acids, suitably sulphuric acid, nitric acid, hydrochloric acid or residual acid from a chlorine dioxide reactor, either separately or in an optional mixture. Preferably, sulphuric acid is employed.

Compounds containing magnesium relate to water-soluble chemicals containing magnesium or mixtures of water-soluble chemicals containing magnesium and calcium. Use is suitably made of magnesium-containing compounds such as magnesium sulphate or magnesium chloride, or calcium-containing compounds such as calcium chloride or calcium oxide. Use is preferably made of magnesium sulphate or magnesium chloride, the use of magnesium sulphate being especially preferred. The combination of temperature and pH at the addition of the compound containing magnesium is always so chosen that the compound is in aqueous solution when contacted with the pulp.

In the process according to the invention, the acid bleaching step is carried out at a pH of from about 1 up to about 6, suitably from 1.5 up to 5, preferably from 2 up to 4. It is especially preferred that the acid bleaching step is carried out at a pH of from 2 up to 3. The magnesium addition is made at a pH in the range from about 1 up to about 7, suitably in the range from 2 up to 6, preferably in the range from 2 up to 4. It is especially preferred that the addition of magnesium is made at a pH of from 2 up to 3. The chlorine-free bleaching agent is hydrogen peroxide and the pulp is treated at a pH of from about 8 up to about 12, preferably at a pH of from 10 up to 12. Treatment with the other chlorine-free bleaching agents mentioned above, is carried out within the normal pH ranges for each bleaching agent, which are well-known to the person skilled in the art.

The treatment according to the invention is carried out with a washing step between the acid bleaching step and the addition of magnesium ions, such that the trace metals that are harmful to the treatment with a chlorine-free bleaching agent are removed from the pulp suspension.

The realization of the process of claim 1 can be carried out at an optional position in the bleaching sequence, e.g. immediately after digestion of the pulp or after an oxygen step. The process according to the invention is preferably applied to pulp that has been delignified in an oxygen step prior to the treatment.

Bleaching and/or delignifying chemicals active within the pH range suitable in the acid treatment are chlorine dioxide, ozone, peracetic acid and/or an acid peroxide-containing compound. Suitably, a combination of acid treatment and bleaching and/or delignifying treatment takes place in an ozone step.

Lignocellulose-containing pulps relate to chemical pulps of softwood and/or hardwood digested according to the sulphite, sulphate, soda or organosolv process, or modifications and/or combinations thereof. Use is suitably made of softwood and/or hardwood digested according to the sulphate process, preferably sulphate pulp of hardwood.

The treatment according to the invention can be applied to lignocellulose-containing pulps having an initial kappa number within the range from about 5 up to about 40, suitably 7 up to 32, preferably from 10 up to 20. Here, the kappa number is determined according to the standard method SCAN-C 1:77.

In the process according to the invention, the acid bleaching step is carried out at a temperature of from about 10 up to about 95°C, suitably from 20 up to 80°C and preferably from 40 up to 80°C, and for a period of time of from about 1 up to about 120 min, suitably from 10 up to 120 min and preferably from 20 up to 40 min. The compound containing magnesium is added at a temperature of from about 10 up to about 95°C, preferably from 40 up to 80°C, and for a

period of time of from about 1 up to about 180 min, preferably from 20 up to 180 min and preferably from 30 up to 120 min. During bleaching with hydrogen peroxide, the pulp is treated at a temperature of from about 30 up to about 100°C, preferably from 60 up to 90°C, and for a period of time of from about 30 up to about 300 min, suitably from 60 up to 240 min. In the acid bleaching step and in the addition of magnesium ions, the pulp concentration may be from about 5 3 up to about 35% by weight, preferably from 3 up to 15% by weight. During bleaching with hydrogen peroxide, the pulp concentration may be from about 3 up to about 50% by weight, suitably from 3 up to 35% by weight and preferably from 10 up to 25% by weight. Treatment with the other chlorine-free bleaching agents mentioned above, is carried out within the normal ranges of temperature, time and pulp concentration for each bleaching agent, which are well-known to the person skilled in the art.

10 The amount of magnesium compound charged, lies in the range from about 0.01 up to about 10 kg/ton of dry pulp, calculated as alkaline earth metal, suitably in the range from 0.5 up to 5 kg/ton of dry pulp, calculated as magnesium, and preferably in the range from 2 up to 4 kg/ton of dry pulp, calculated as magnesium.

15 The amount of hydrogen peroxide, lies in the range from about 2 up to about 50 kg/ton of dry pulp, calculated as 100% hydrogen peroxide. The upper limit is not critical, but has been set for reasons of economy. The amount of hydrogen peroxide suitably lies in the range from 3 up to 30 kg/ton of dry pulp and preferably from 4 up to 20 kg/ton of dry pulp, calculated as 100% hydrogen peroxide.

20 After the completion of the process of claim 1, the pulp can be used for direct production of paper with a lower demand of brightness. Alternatively, the pulp may be finally bleached to the desired higher brightness, by treatment in one or more steps. Suitably, the final bleaching is also carried out with chlorine-free bleaching agents of the type 25 mentioned above, optionally with intermediate alkaline extraction steps, which may be reinforced with peroxide and/or oxygen. In this way, the formation and discharge of AOX is completely eliminated. Suitably, the final bleaching is carried out with ozone in one or more steps. By the treatment according to the invention, the lignin content has been reduced to a sufficiently low level before any chlorine-containing bleaching agents are used. Therefore, chlorine dioxide and/or hypochlorite may well be used in one or more final bleaching steps without causing formation of large amounts of AOX.

30 Moreover, use of the process according to the invention means that the brightness and kappa number of the resulting pulp is higher and lower, respectively, than with the processes in which a compound containing an alkaline earth metal is not added at all or is added at a higher pH. In a process for bleaching chemical pulps, the aim is a high brightness as well as a low kappa number, the latter meaning a low content of undissolved lignin. At the same time, the consumption of the chlorine-free bleaching agent should be as low as possible meaning lower treatment costs. In the process according to the invention, these objects are met, as is apparent from the Examples. Furthermore, the strength of the pulp, measured as viscosity, is sufficient, which means that the pulp contains cellulose chains which are long enough to give a strong product.

35 The invention and its advantages are illustrated in more detail by the Examples below. The percentages and parts stated in the description, claims and examples, refer to percent by weight and parts by weight, respectively, unless anything else is stated.

Example 1

40 Oxygen-delignified sulphate pulp of softwood having a kappa number of 13.7, a brightness of 37.1% ISO and a viscosity of 1057 dm³/kg, was treated in the sequences D - EOP-Z - P (test 1) and D - EOP - Z - Mg - P (test 2), where the conditions in each step were equal in both sequences. D and EOP relate to a conventional chlorine dioxide step and a conventional alkaline extraction step reinforced with hydrogen peroxide and oxygen, respectively. Z relates to 45 an ozone step with a pH of 2.3. Mg relates to the addition of 1 kg of magnesium/ton of dry pulp, in the form of an aqueous solution containing MgSO₄. Magnesium was added at a pH of 4.1, a temperature of 50°C for 30 min, the pulp concentration being about 3% by weight. P relates to a hydrogen peroxide step, where the pulp was treated at a temperature of 80°C for 120 min. The final pH was about 11.5 and the addition of hydrogen peroxide was 5 kg/ton of dry pulp calculated as 100% hydrogen peroxide. The viscosity and brightness were determined according to SCAN Standard Methods. The results after bleaching with hydrogen peroxide appear from the Table below.

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TABLE I

Test	Amount of Mg added (kg/ton)	Amount of Mg in pulp after addition (ppm)	Viscosity (dm ³ /kg)	Brightness (% ISO)
1	0	16	820	86.3
2	1.0	255	872	88.4

As is apparent from the Table, addition of dissolved magnesium after an initial acid treatment with ozone and

- 8. A process according to claim 1, **characterised** in that the bleaching before addition of a water-soluble chemical containing magnesium is carried out at a pH in the range from 1.5 up to 5.
- 5 9. A process according to claim 1, **characterised** in that the pulp, after the bleaching with hydrogen peroxide at a pH of from about 8 up to about 12, is finally bleached with ozone in one or more steps.
- 10 10. A process according to claims 1-9, **characterised** in that the acid bleaching step is carried out at a temperature of from about 10 up to about 95°C for about 1 up to about 120 min, that the water-soluble chemical containing magnesium is added at a temperature of from about 10 up to 95°C for about 1 up to about 180 min and in an amount of from 0.5 up to 5 kg/ton of dry pulp, calculated as magnesium, the treated pulp having a concentration of from about 3 up to about 35% by weight.

Patentansprüche

- 15 1. Verfahren zum Entholzen und Bleichen von chemisch aufgeschlossenem lignocellulosehaltigem Zellstoff, dadurch gekennzeichnet, daß der Zellstoff bei einem pH-Wert im Bereich von etwa 1 bis etwa 6 mit einer aus Chlordioxid, Ozon, Peressigsäure und Säureperoxiden ausgewählten Bleichchemikalie gebleicht wird, wonach der Zellstoff gewaschen wird, worauf eine Magnesium enthaltende wasserlösliche Chemikalie bei einem pH-Wert im Bereich von etwa 1 bis etwa 7 und in einer Menge von etwa 0.01 bis etwa 10 kg/Tonne trockener Zellstoff, berechnet als Magnesium, zugegeben wird, und daß der Zellstoff anschließend bei einem pH-Wert von etwa 8 bis etwa 12 mit Wasserstoffperoxid gebleicht wird.
- 20 2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Zellstoff anschließend bei einem pH-Wert von 10 bis 12 mit Wasserstoffperoxid gebleicht wird.
- 25 3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der chemisch aufgeschlossene Zellstoff Sulfatzellstoff ist.
- 30 4. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Magnesium enthaltende wasserlösliche Chemikalie bei einem pH-Wert von 2 bis 6 zugegeben wird.
- 35 5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Magnesium enthaltende wasserlösliche Chemikalie ein Gemisch aus magnesiumhaltigen und calciumhaltigen Verbindungen ist.
- 6. Verfahren nach Anspruch 1 oder 5, dadurch gekennzeichnet, daß die Magnesium enthaltende wasserlösliche Chemikalie aus Magnesiumsulfat oder Magnesiumchlorid besteht.
- 40 7. Verfahren nach Anspruch 5, dadurch gekennzeichnet, daß die Calcium enthaltende wasserlösliche Chemikalie aus Calciumchlorid oder Calciumoxid besteht.
- 8. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das Bleichen vor der Zugabe einer Magnesium enthaltenden wasserlöslichen Chemikalie bei einem pH-Wert im Bereich von 1.5 bis 5 durchgeführt wird.
- 45 9. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Zellstoff nach dem Bleichen mit Wasserstoffperoxid bei einem pH-Wert von etwa 8 bis etwa 12 schließlich in einem oder mehreren Schritten mit Ozon gebleicht wird.
- 10. Verfahren nach den Ansprüchen 1-9, dadurch gekennzeichnet, daß der Schritt der Säurebleichung bei einer Temperatur von etwa 10 bis etwa 95°C während etwa 1 bis etwa 120 Min. durchgeführt wird, daß die Magnesium enthaltende wasserlösliche Chemikalie bei einer Temperatur von etwa 10 bis 95°C während etwa 1 bis etwa 180 Min. und in einer Menge von 0.5 bis 5 kg/Tonne trockener Zellstoff, berechnet als Magnesium, zugegeben wird, wobei der behandelte Zellstoff eine Konzentration von etwa 3 bis etwa 35 Gew.-% hat.

Revendications

- 1. Procédé de délignification et de blanchiment d'une pâte à papier contenant de la lignocellulose et obtenue par digestion chimique, caractérisé en ce que l'on blanchit la pâte, à un pH situé dans l'intervalle allant d'environ 1 à

bleaching with hydrogen peroxide according to the present invention, positively influence the pulp as regards viscosity and brightness.

Example 2

The oxygen-delignified sulphate pulp of softwood used in Example 1, was treated in the sequences Step1 - P₁ - Z - P₂ (test 1) and Step1 - P₁ - Z - Mg - P₂ (test 2), where the conditions in each step were equal in both sequences. Step1 relates to treatment with EDTA at a pH of 5. Z relates to an ozone step with a pH of 2.3, the pulp concentration being 10% by weight. Mg relates to the addition of 1 kg of magnesium/ton of dry pulp, in the form of an aqueous solution containing MgSO₄. Magnesium was added at a pH of 4.1, a temperature of 50°C for 30 min, the pulp concentration being about 3% by weight. P₂ relates to a hydrogen peroxide step, where the pulp was treated at a temperature of 80°C for 120 min. The final pH was about 11.5 and the addition of hydrogen peroxide was 5 kg/ton of dry pulp calculated as 100% hydrogen peroxide. For comparative purposes, the pulp was also treated in the sequence Step1 - P₁ - Z - (PMg) (test 3). (PMg) relates to the addition of magnesium in the second alkaline hydrogen peroxide step under the conditions stated above, in accordance with the prior art. The viscosity and brightness were determined according to SCAN Standard Methods, and the consumption of hydrogen peroxide was determined by iodometric titration. The results after the second hydrogen peroxide step appear from the Table below.

TABLE II

Test	Amount of Mg added (kg/ton)	Amount of Mg in pulp (ppm)	Viscosity (dm ³ /kg)	Brightness (% ISO)	H ₂ O ₂ consumed in P ₂ (% of added)
1	0	9	794	80.9	76
2	1.0	300	856	83.9	28
3	1.0	120	809	81.6	60

As is apparent from the Table, addition of magnesium within the present pH range before bleaching with hydrogen peroxide, positively influence the pulp as regards viscosity and brightness and reduces the consumption of hydrogen peroxide.

Claims

1. A process for delignification and bleaching of a chemically digested lignocellulose-containing pulp, characterised in that the pulp is bleached at a pH in the range from about 1 up to about 6 with a bleaching chemical selected from the group consisting of chlorine dioxide, ozone, peracetic acid and acid peroxides, thereafter the pulp is washed, whereupon a water-soluble chemical containing magnesium is added at a pH in the range from about 1 up to about 7 and in an amount of from about 0.01 up to about 10 kg/ton of dry pulp, calculated as magnesium, and that subsequently the pulp is bleached with hydrogen peroxide at a pH of from about 8 up to about 12.
2. A process according to claim 1, characterised in that subsequently the pulp is bleached with hydrogen peroxide, at a pH of from 10 up to 12.
3. A process according to claim 1, characterised in that the chemically digested pulp is a sulphate pulp.
4. A process according to claim 1, characterised in that water-soluble chemical containing magnesium is added at a pH of from 2 up to 6.
5. A process according to claim 1, characterised in that the water-soluble chemical containing magnesium is a mixture of magnesium-containing and calcium-containing compounds.
6. A process according to claim 1 or 5, characterised in that the water-soluble chemical containing magnesium consists of magnesium sulphate or magnesium chloride.
7. A process according to claim 5, characterised in that the water-soluble chemical containing calcium consists of calcium chloride or calcium oxide.

environ 6, à l'aide d'un agent de blanchiment chimique choisi dans l'ensemble que constituent le dioxyde de chlore, l'ozone, l'acide peracétique et les peroxydes d'acide, puis on lave la pâte, après quoi l'on ajoute, à un pH situé dans l'intervalle allant d'environ 1 à environ 7, un produit chimique hydrosoluble contenant du magnésium, en une quantité, calculée en magnésium, d'environ 0,01 à environ 10 kg par tonne de pâte sèche, et l'on blanchit ensuite la pâte avec du peroxyde d'hydrogène, à un pH valant d'environ 8 à environ 12.

- 5 2. Procédé conforme à la revendication 1, caractérisé en ce que l'on blanchit ensuite la pâte avec du peroxyde d'hydrogène, à un pH valant de 10 à 12.
- 10 3. Procédé conforme à la revendication 1, caractérisé en ce que la pâte obtenue par digestion chimique est une pâte au sulfate.
- 15 4. Procédé conforme à la revendication 1, caractérisé en ce que l'addition du produit chimique hydrosoluble contenant du magnésium est effectuée à un pH de 2 à 6.
- 5 5. Procédé conforme à la revendication 1, caractérisé en ce que le produit chimique hydrosoluble contenant du magnésium est un mélange de composés contenant du calcium et du magnésium.
- 20 6. Procédé conforme à la revendication 1 ou 5, caractérisé en ce que le produit chimique hydrosoluble contenant du magnésium est du sulfate de magnésium ou du chlorure de magnésium.
7. Procédé conforme à la revendication 5, caractérisé en ce que le produit chimique hydrosoluble contenant du calcium est du chlorure de calcium ou de l'oxyde de calcium.
- 25 8. Procédé conforme à la revendication 1, caractérisé en ce que le blanchiment réalisé avant l'addition du produit chimique hydrosoluble contenant du magnésium est effectué à un pH de 1,5 à 5.
9. Procédé conforme à la revendication 1, caractérisé en ce que la pâte, après avoir été blanchie à l'aide de peroxyde d'hydrogène à un pH d'environ 8 à environ 12, est finalement blanchie avec de l'ozone, en une ou plusieurs étapes.
- 30 10. Procédé conforme à l'une des revendications 1 à 9, caractérisé en ce que l'étape de blanchiment en milieu acide est effectuée à une température d'environ 10°C à environ 95°C et pendant d'environ 1 à environ 120 minutes, et en ce que le produit chimique hydrosoluble contenant du magnésium est ajouté à une température d'environ 10°C à environ 95°C, en un laps de temps d'environ 1 à environ 180 minutes et en une quantité, calculée en magnésium, de 0,5 à 5 kg par tonne de pâte sèche, la concentration de la pâte traitée valant d'environ 3 à environ 35 % en poids.

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